

PATENT ABSTRACTS OF JAPAN

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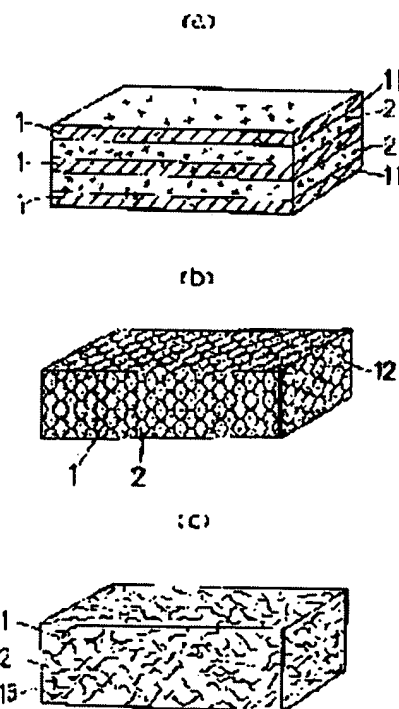
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(22)Date of filing : 27.07.1994 (72)Inventor : SONODA KENJI
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(54) AEROGEL PANEL

(57)Abstract:

PURPOSE: To obtain an aerogel panel with improved strength having translucency while maintaining heat insulating property by using a fiber body having heat insulating property as the core material and depositing an aerogel having a silica skeleton on the core material.

CONSTITUTION: As for the fiber material 1 having heat insulating property as the core material, a multilayered nonwoven fabric (a) having translucency, an aggregated fiber block (b) having voids or dispersed fibers (c) is used. The core is impregnated with an alkoxysilane sol comprising alkoxysilane, water and alcohol. After the sol is changed into a gel, the material is dried under supercritical conditions higher than the critical point of the solvent to produce an aerogel. Thus, the aerogel 2 having a silica skeleton deposits on the fiber 1, and thereby, an aerogel panel in such a state that the aerogel 2 is reinforced with the fiber material 1 is obtd.



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CLAIMS

[Claim(s)]

[Claim 1] The aerogel panel characterized by consisting of aerogel (2) which has the silica frame to which the fiber object (1) which has adiathermic as a core material, and this fiber object (1) adhered.

[Claim 2] The aerogel panel according to claim 1 characterized by the above-mentioned fiber object (1) consisting of a multilayer nonwoven fabric which has translucency.

[Claim 3] The aerogel panel according to claim 1 characterized by the above-mentioned fiber object (1) consisting of the massive fiber aggregate which has an opening.

[Claim 4] The aerogel panel according to claim 1 characterized by consisting of fiber which the above-mentioned fiber object (1) distributed.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the aerogel panel which has adiathermic.

[0002]

[Description of the Prior Art] The aerogel which has a silica frame as an insulator is known. This aerogel is obtained by drying the gel compound of the damp or wet condition which consists of a silica frame which hydrolyzes alkoxysilane (it is also independently called a silicon alkoxide and alkyl silicate), and is obtained by carrying out condensation polymerization on the supercritical conditions more than the critical point of this solvent under existence of solvents, such as alcohol or a liquefaction carbon dioxide, as indicated by U.S. Pat. No. 4402927, U.S. Pat. No. 4432956, and U.S. Pat. No. 4610863. This aerogel is useful as a heat insulator which has translucency. However, since reinforcement is small and weak, it is divided, or the aerogel obtained by these approaches tends to break, and has a fault with difficult handling, while it is very lightweight.

[0003]

[Problem(s) to be Solved by the Invention] The place which this invention was made in view of the above-mentioned fact, and is made into the purpose maintains adiathermic, and is to offer the aerogel panel whose reinforcement improved.

[0004] Furthermore, the place made into other purposes is to offer the aerogel panel which has translucency.

[0005]

[Means for Solving the Problem] The aerogel panel concerning claim 1 of this invention is characterized by consisting of aerogel 2 which has the silica frame to which the fiber object 1 which has adiathermic as a core material, and this fiber object 1 adhered.

[0006] The aerogel panel concerning claim 2 of this invention is characterized by the above-mentioned fiber object 1 consisting of a multilayer nonwoven fabric which has translucency in an aerogel panel according to claim 1.

[0007] The aerogel panel concerning claim 3 of this invention is characterized by the above-mentioned fiber object 1 consisting of the massive fiber aggregate which has an opening in an aerogel panel according to claim 1.

[0008] The aerogel panel concerning claim 4 of this invention is characterized by consisting of fiber which the above-mentioned fiber object 1 distributed in an aerogel panel according to claim 1.

[0009] This invention is explained to a detail based on a drawing below. Drawing 1 (a), (b), and (c) are the perspective views of the aerogel panel concerning one example of this invention, and drawing 2, drawing 3, drawing 4, and drawing 5 are the schematic diagrams having shown an example of a process which obtains an aerogel panel.

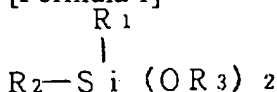
[0010] The aerogel panel of this invention consists of aerogel 2 which has the silica frame to which the fiber object 1 and this fiber object 1 adhered as a core material. The above-mentioned aerogel 2 is a porous material obtained by drying the gel compound of the damp or wet condition which consists of a silica frame which hydrolyzed the alkoxysilane compound and was obtained by carrying out condensation polymerization on the supercritical conditions more than the critical point of this solvent under existence of solvents, such as alcohol or a liquefaction carbon dioxide.

[0011] As the above-mentioned alkoxysilane compound used for this invention, the oligomer of 2 organic-functions alkoxysilane expressed with a bottom type (** 1), 3 organic-functions alkoxysilane expressed with a bottom type (** 2), 4 organic-functions alkoxysilane expressed with a bottom type (** 3), and the alkoxysilane

expressed with a bottom type (** 4) is mentioned.

[0012]

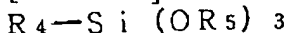
[Formula 1]



[0013] R1, R2, and R3 show the alkyl group or phenyl group of carbon numbers 1-5 mutually-independent among [type. Two R3 It may be mutually the same and you may differ.]

[0014]

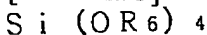
[Formula 2]



[0015] R4 and R5 show the alkyl group or phenyl group of carbon numbers 1-5 mutually-independent among [type. Three R5 It may be mutually the same and you may differ.]

[0016]

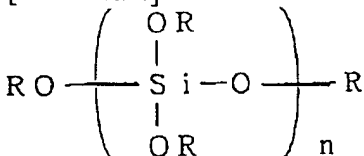
[Formula 3]



[0017] R6 shows the alkyl group or phenyl group of carbon numbers 1-5 among [type. Four R6 It may be mutually the same and you may differ.]

[0018]

[Formula 4]



[0019] R shows the alkyl group or phenyl group of carbon numbers 1-5 among [type, and n is an integer which shows polymerization degree. However, as long as it is in a structurally stable condition until it starts hydrolysis and condensation polymerization, some OR radicals may be permuted by the OH radical.]

Specifically as the above-mentioned 2 organic-functions alkoxysilane, dimethyl dimethoxysilane, dimethyl diethoxysilane, diphenyl diethoxysilane, diphenyl dimethoxysilane, methylphenyl diethoxysilane, methylphenyl dimethoxysilane, diethyl diethoxysilane, diethyl dimethoxysilane, etc. are mentioned.

[0020] Specifically as the above-mentioned 3 organic-functions alkoxysilane, methyl trimethoxysilane, methyl triethoxysilane, ethyl trimethoxysilane, ethyltriethoxysilane, phenyl trimethoxysilane, phenyl triethoxysilane, etc. are mentioned.

[0021] Specifically as the above-mentioned 4 organic-functions alkoxysilane, a tetramethoxy silane, a tetraethoxy silane, etc. are mentioned.

[0022] transparent and colorless, although it is desirable that polymerization degree is below ten (polymerization degree describes the thing of n as n **** below.) as for the oligomer of the alkoxysilane expressed above (** 4) -- if liquefied, it will not be limited to this. The oligomer of the above-mentioned alkoxysilane does not need to be a compound with this uniform polymerization degree, distribution of polymerization degree may exist or the molecular structure may be intermingled by the shape of a chain, the letter of branching, and annular. If the stability as matter and the reaction time for producing a gel compound are taken into consideration, the thing of 2 - a hexamer is the most desirable. R in the oligomer of the above-mentioned alkoxysilane expresses an alkyl group and a phenyl group, and a methyl group (CH3) and its ethyl group (C two H5) are the most desirable especially. In the case of the oligomer of a methoxy silane, when average molecular weight is OGORIMA of 250-700, and an ethoxy silane, specifically, the oligomer of 300-900 has the most desirable average molecular weight.

[0023] In order to hydrolyze the above-mentioned alkoxysilane efficiently and to perform condensation polymerization, it is desirable to add a catalyst beforehand to the system of reaction containing alkoxysilane. As the above-mentioned catalyst, basic catalysts, such as an acid catalyst of a hydrochloric acid, a citric acid, a nitric acid, a sulfuric acid, ammonium fluoride, etc. and ammonia, and a piperidine, are mentioned, for example.

[0024] Furthermore, in order to obtain a uniform gel compound, it is desirable to use mixed liquor with the solvent which has compatibility in water and water and dissolves the above-mentioned alkoxysilane in the above-mentioned system of reaction. As the above-mentioned solvent, alcohol and acetones, such as a methanol, ethanol, isopropanol, and a butanol, an acetonitrile, etc. are mentioned, for example. Alcohol is the most desirable when alcohol's generating at hydrolysis of the generation process of a gel compound and a polymerization reaction and the supercritical drying mentioned later are taken into consideration.

[0025] The fiber object 1 which constitutes the aerogel panel of this invention has adiathermic, and organic high molecular compounds, such as glass fiber, ceramic fiber, polyester, and a polyamide, are mentioned as the quality of the material. These fiber object 1 is used after fiber has only distributed, the multilayer object of a nonwoven fabric, the aggregate of massive fiber, and. By using the fiber object 1 for this core material, the reinforcement of aerogel 2 is reinforced, and aerogel 2 breaks in the case of handling, or it prevents being destroyed. Furthermore, adiathermic [of aerogel] is maintainable. The class of fiber object 1 used as the above-mentioned core material, a configuration, a consistency, thickness, etc. are suitably chosen from the physical properties which the aerogel panel obtained needs.

[0026] Next, the manufacture approach for obtaining the above-mentioned aerogel panel is explained. In order to obtain the above-mentioned aerogel panel, after making the sol-like reaction mixture 3 containing alkoxysilane, the above-mentioned solvent, etc. adhere to the above-mentioned fiber object 1 and gelling it, supercritical drying is carried out and it produces.

[0027] An example which obtains the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) is shown. The nonwoven fabric 11 which has translucency as a fiber object 1 is used. For thickness, 0.1-0.5mm and a consistency are [the above-mentioned nonwoven fabric 11] 0.1 - 2.0 g/cm³. It is desirable. The translucency of the above-mentioned nonwoven fabric 11 is suitably chosen by the application of an aerogel panel. As shown in drawing 2, after covering the bottom of a container 4 with one nonwoven fabric 11a and slushing the above-mentioned sol-like reaction mixture 3a on this nonwoven fabric 11a Before this slushed sol-like reaction mixture 3 gels, nonwoven fabric 11b is floated on the top face of this sol-like reaction mixture 3, and after the above-mentioned sol-like reaction mixture 3a which carried out the style gels, sol-like reaction mixture 3b is slushed on nonwoven fabric 11b of the 2nd sheet from a bottom. By repeating until the thickness of a request of this is obtained, the layered product which sol-like reaction mixture 3 gelled between the layers of a nonwoven fabric 11 is obtained. If supercritical drying of this layered product is carried out, the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) will be obtained.

[0028] Moreover, as shown in drawing 3, the layered product which sol-like reaction mixture 3 gelled between the layers of a nonwoven fabric 11 is obtained by opening two or more sheets and spacing 5 in the bottom of a container 4, installing a nonwoven fabric 11 in it in the cross direction, and making this spacing 5 slush and gel the above-mentioned sol-like reaction mixture 3. If supercritical drying of this layered product is carried out, the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) will be obtained. Since the obtained aerogel panel uses the nonwoven fabric 11 which has translucency, its translucency is good, while its reinforcement improves and it maintains adiathermic.

[0029] An example which obtains the aerogel panel which used as the fiber object 1 the massive fiber aggregate 12 shown in drawing 1 (b) is shown. As a fiber object 1, the massive fiber aggregate 12 which has openings, such as glass wool, is used. For the above-mentioned massive fiber aggregate 12, a consistency is 0.005 - 0.5 g/cm³. The range is desirable. As shown in drawing 4, the massive fiber aggregate 12 of desired magnitude is put into the container 4, the above-mentioned sol-like reaction mixture 3 is slushed in a container 4, and the opening of the above-mentioned massive fiber aggregate 12 is filled up with sol-like reaction mixture 3. After the above-mentioned sol-like reaction mixture 3 gels, if supercritical drying is performed, the aerogel panel which used as the fiber object 1 the massive fiber aggregate 12 shown in drawing 1 (b) will be obtained.

[0030] An example which obtains the aerogel panel used as the fiber object 1 which consists of dispersed fiber 13 shown in drawing 1 (c) is shown. Fiber 13 with a diameter [of 1-100 micrometers] and a die length of about 1-5cm is used. It slushes into a container 4, after carrying out mixed stirring of the above-mentioned sol-like reaction mixture 3 and the above-mentioned fiber 13 beforehand, as shown in drawing 5. The loadings of the above-mentioned fiber 13 have 5 - 30 desirable % of the weight to the aerogel panel obtained. After the above-mentioned sol-like reaction mixture 3 gels, if supercritical drying is performed, the aerogel panel used as the

fiber object 1 which consists of dispersed fiber 13 shown in drawing 1 (c) will be obtained.

[0031] When hydrophobing processing is performed in the case of supercritical drying before the above-mentioned supercritical drying, degradation of the dimensional change by moisture absorption of an aerogel panel etc. can be prevented, and it is desirable. The above-mentioned hydrophobing processing permutes the hydrophilic group on the front face of a silica by the hydrophobic group, and uses the hydrophobing processing agent which has the functional group which reacts to a silanol group, and a hydrophobic group. Organic silane compounds, such as hexamethyldisilazane, hexa methyl disiloxane, trimethylchlorosilane, a trimethyl methoxy silane, a trimethyl ethoxy silane, a triethyl ethoxy silane, a triethyl methoxy silane, dimethyldichlorosilane, dimethyl diethoxysilane, methyltrichlorosilane, and ethyl trichlorosilane, are mentioned, and, specifically, as for the above-mentioned hydrophobing processing agent, organic compounds, such as carboxylic acids, such as an acetic acid, formic acid, and a succinic acid, and alkyl halides, such as methyl chloride, are mentioned besides this. A hydrophobing processing agent may use only one sort and may use two or more sorts.

[0032] As a solvent used in case supercritical drying of the fiber object 1 to which the above-mentioned gel compound adhered is carried out, independent or two or more sorts of mixed stock, such as ethanol, a methanol, a dichlorodifluoromethane, a carbon dioxide, and water, are mentioned, for example.

[0033] When using the above-mentioned solvent independently, generally, the gel compound which performed the solvent permutation is put into the above-mentioned solvent and the same solvent together into proof-pressure containers, such as an autoclave, the inside of a container is raised to the temperature more than the critical point of this solvent, and a pressure, the solvent contained in the gel compound is removed gradually, and, finally desiccation is ended by [of the condition of ordinary temperature ordinary pressure] returning. When using the above-mentioned solvent by two or more sorts of mixing, the solvent contained in a gel compound dissolves in the solvent mixed within the proof-pressure container. The above-mentioned gel compound is made to coexist in the same solvent as the approach of raising a pressure and temperature so that it may be in a supercritical condition in the state of 1 phase, and the solvent contained in a gel compound within a proof-pressure container. Since the solvent in a gel compound is permuted by another soluble high solvent in the state of high pressure and the permutation of a solvent is completed mostly, it changes into the supercritical condition of the above-mentioned solvent, and there is a method of removing the solvent contained in the gel compound etc.

[0034]

[Function] The fiber object 1 reinforces the reinforcement of aerogel 2 as a core material, aerogel 2 breaks in the case of handling, or it prevents being destroyed while it maintains adiabatic, since the aerogel panel concerning claim 1 of this invention consists of aerogel 2 which has the silica frame to which the fiber object 1 which has adiabatic as a core material, and this fiber object 1 adhered.

[0035]

[Example]

The oligomer (COL coat incorporated company make: methyl silicate 51, average molecular weight 470) of a tetramethoxy silane was used as example 1 alkoxysilane, and 0.01 mols [1.] aqueous ammonia was used as a solvent as ethanol (reagent chemicals by Nakarai Tesuku, Inc.), water, and a catalyst. The sol-like reaction mixture which blended 120 mols and water by 20 mols, and blended [the oligomer of the above-mentioned tetramethoxy silane] aqueous ammonia for one mol and ethanol by the ratio of 2.16 mols was obtained.

[0036] As a fiber object, the nonwoven fabric (the Kureha tech incorporated company make: die nak S-020) of 0.15mm in thickness and specific gravity 1.08 was used. The bottom of a container was covered with one sheet of this nonwoven fabric, and the above-mentioned sol-like reaction mixture was slushed. After it floated the nonwoven fabric of the 2nd sheet on the sol-like reaction mixture slushed after [of abbreviation] 2 minutes and previous sol-like reaction mixture gelled, sol-like reaction mixture was slushed on the nonwoven fabric of the 2nd sheet. Floated the nonwoven fabric of the 3rd sheet on the sol-like reaction mixture slushed after [of abbreviation] 2 minutes, it was made to put and gel at a room temperature, and the layered product of a gel compound and a nonwoven fabric was obtained. The thickness of this layered product was 8mm.

[0037] Next, a proof-pressure container is used and they are 18 degrees C and the pressure of 55kg/cm² about this layered product. It put in into the liquefaction carbon dioxide and actuation of permuting the ethanol in a gel compound by the carbon dioxide was performed for 3 hours. The temperature of 80 degrees C and the pressure of 160kg/cm² which are the supercritical conditions of a carbon dioxide about the inside of a proof-pressure container after that It carried out and solvent removal was performed for 48 hours. Add

hexamethyldisilazane in the ambient atmosphere of this supercritical condition at a rate of 0.3 mols/l. as a hydrophobing processing agent, and it was made to diffuse a hydrophobing processing agent in supercritical fluid over 2 hours, into this supercritical fluid, the gel compound was left and hydrophobing was given. Then, it decompressed, after circulating the carbon dioxide of a supercritical condition, and the ethanol and the hydrophobing processing agent which are contained in a gel compound were removed. The time amount from a hydrophobing processing agent injection to reduced pressure required 15 hours. Then, it took out from the proof-pressure container and the aerogel panel was obtained. this aerogel panel -- thickness -- 8mm and bulk density -- 0.086 g/cm³ it was .

[0038] The aerogel panel was obtained like the example 1 except having obtained the layered product with a thickness of 8.0mm which made three layers the gel compound of example 2 example 1, and made the nonwoven fabric four layers. this aerogel panel -- thickness -- 8mm and bulk density -- 0.115 g/cm³ it was .

[0039] As an example 3 fiber object, they are 0.8mm in thickness, and consistency 0.01 g/cm³. Glass wool (NIPPON MUKI [CO., LTD.] make: super fine SPF-210) was used. After putting this glass wool into a container and slushing the same sol-like reaction mixture as an example 1, it was made to put and gel at a room temperature. Supercritical drying was performed on the same conditions as an example 1 after that, and the aerogel panel was obtained. this aerogel panel -- thickness -- 8mm and bulk density -- 0.055 g/cm³ it was .

[0040] In example 4 example 3, the aerogel panel was obtained like the example 3 except having not performed hydrophobing processing on the occasion of supercritical drying. this aerogel panel -- thickness -- 8mm and bulk density -- 0.051 g/cm³ it was .

[0041] The oligomer (COL coat incorporated company make: methyl silicate 51, average molecular weight 470) of a tetramethoxy silane was used as example 5 alkoxysilane, and 0.01 mols [/l.] aqueous ammonia was used as a solvent as ethanol (reagent by Nakarai Tesuku, Inc.), water, and a catalyst. The sol-like reaction mixture which blended 50 mols and water by 20 mols, and blended [the oligomer of the above-mentioned tetramethoxy silane] aqueous ammonia for one mol and ethanol by the ratio of 0.22 mols was obtained. The aerogel panel was obtained like the example 3 except having used the above-mentioned sol-like reaction mixture. this aerogel panel -- thickness -- 8mm and bulk density -- 0.121 g/cm³ it was .

[0042] The same sol-like reaction mixture as example 6 example 1 was used. Fiber with a die length of 20mm was mixed and stirred at a rate of the 0.1 weight section and the above-mentioned sol-like reaction mixture 14 weight section, and it slushed into the container. Supercritical drying was carried out on the same conditions as an example 1 after gelation, and the aerogel panel was obtained. The rate of the above-mentioned fiber became 12 % of the weight to the obtained aerogel panel. moreover, this aerogel panel -- thickness -- 8mm and bulk density -- 0.051 g/cm³ it was .

[0043] After obtaining a gel compound using the same sol-like reaction mixture as example of comparison 1 example 1, supercritical drying was carried out on the same conditions as an example 1, and aerogel was obtained. this aerogel -- thickness -- 8mm and bulk density -- 0.045 g/cm³ it was .

After obtaining a gel compound using the same sol-like reaction mixture as example of comparison 2 example 5, supercritical drying was carried out on the same conditions as an example 5, and aerogel was obtained. this aerogel -- thickness -- 8mm and bulk density -- 0.085 g/cm³ it was .

[0044] It evaluated the aerogel panel of the acquired examples 1-6, the reinforcement of the aerogel of the examples 1-2 of a comparison, and adiathermic. Flexural strength was measured as reinforcement. Thermal conductivity was measured as adiathermic. The above-mentioned thermal conductivity measured the thermal conductivity of 20 degrees C - 40 degrees C based on ASTM-C518 using the Hidehiro energy machine incorporated company make and thermal conductivity measuring apparatus.

[0045] As for the example, adiathermic was maintained and it was checked that reinforcement is improving as the result was shown in Table 1.

[0046]

[Table 1]

	曲げ強度 (kg/cm ²)	熱伝導率 (Kcal/mh℃)
実施例 1	2.40	0.014
実施例 2	3.18	0.015
実施例 3	1.86	0.016
実施例 4	1.77	0.016
実施例 5	3.56	0.014
実施例 6	0.32	0.013
比較例 1	0.05	0.013
比較例 2	0.09	0.010

[0047] The light transmittance of the aerogel panel of the acquired examples 1, 2, and 6 and the aerogel of the examples 1-2 of a comparison was measured. The above-mentioned light transmittance measured the spectral distribution of a light region, and asked for light permeability based on JIS-R3106. It was checked by the result as Table 2 that the aerogel panel of examples 1, 2, and 6 has translucency.

[0048]

[Table 2]

	光透過率 (%)
実施例 1	70
実施例 2	50
実施例 6	81
比較例 1	92
比較例 2	93

[0049]

[Effect of the Invention] The aerogel panel concerning claim 1 thru/or claim 4 of this invention maintains adiathermic, and reinforcement is excellent.

[0050] Furthermore, especially the aerogel panel concerning claim 2 of this invention or claim 4 has translucency.

[0051] The aerogel panel of this invention is useful for applications, such as a heat insulator, an acoustic material, and catalyst support.

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TECHNICAL FIELD

[Industrial Application] This invention relates to the aerogel panel which has adiathermic.

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PRIOR ART

[Description of the Prior Art] The aerogel which has a silica frame as an insulator is known. This aerogel is obtained by drying the gel compound of the damp or wet condition which consists of a silica frame which hydrolyzes alkoxysilane (it is also independently called a silicon alkoxide and alkyl silicate), and is obtained by carrying out condensation polymerization on the supercritical conditions more than the critical point of this solvent under existence of solvents, such as alcohol or a liquefaction carbon dioxide, as indicated by U.S. Pat. No. 4402927, U.S. Pat. No. 4432956, and U.S. Pat. No. 4610863. This aerogel is useful as a heat insulator which has translucency. However, since reinforcement is small and weak, it is divided, or the aerogel obtained by these approaches tends to break, and has a fault with difficult handling, while it is very lightweight.

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EFFECT OF THE INVENTION

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] The place which this invention was made in view of the above-mentioned fact, and is made into the purpose maintains adiathermic, and is to offer the aerogel panel whose reinforcement improved.

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MEANS

[Means for Solving the Problem] The aerogel panel concerning claim 1 of this invention is characterized by consisting of aerogel 2 which has the silica frame to which the fiber object 1 which has adiabatic as a core material, and this fiber object 1 adhered.

[0006] The aerogel panel concerning claim 2 of this invention is characterized by the above-mentioned fiber object 1 consisting of a multilayer nonwoven fabric which has translucency in an aerogel panel according to claim 1.

[0007] The aerogel panel concerning claim 3 of this invention is characterized by the above-mentioned fiber object 1 consisting of the massive fiber aggregate which has an opening in an aerogel panel according to claim 1.

[0008] The aerogel panel concerning claim 4 of this invention is characterized by consisting of fiber which the above-mentioned fiber object 1 distributed in an aerogel panel according to claim 1.

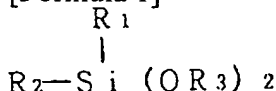
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[0010] The aerogel panel of this invention consists of aerogel 2 which has the silica frame to which the fiber object 1 and this fiber object 1 adhered as a core material. The above-mentioned aerogel 2 is a porous material obtained by drying the gel compound of the damp or wet condition which consists of a silica frame which hydrolyzed the alkoxysilane compound and was obtained by carrying out condensation polymerization on the supercritical conditions more than the critical point of this solvent under existence of solvents, such as alcohol or a liquefaction carbon dioxide.

[0011] As the above-mentioned alkoxysilane compound used for this invention, the oligomer of 2 organic-functions alkoxysilane expressed with a bottom type (** 1), 3 organic-functions alkoxysilane expressed with a bottom type (** 2), 4 organic-functions alkoxysilane expressed with a bottom type (** 3), and the alkoxysilane expressed with a bottom type (** 4) is mentioned.

[0012]

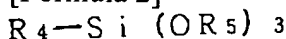
[Formula 1]



[0013] R1, R2, and R3 show the alkyl group or phenyl group of carbon numbers 1-5 mutually-independent among [type. Two R3 It may be mutually the same and you may differ.]

[0014]

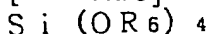
[Formula 2]



[0015] R4 and R5 show the alkyl group or phenyl group of carbon numbers 1-5 mutually-independent among [type. Three R5 It may be mutually the same and you may differ.]

[0016]

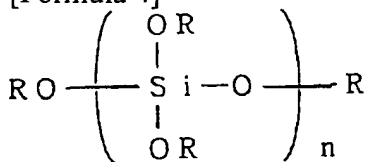
[Formula 3]



[0017] R6 shows the alkyl group or phenyl group of carbon numbers 1-5 among [type. Four R6 It may be mutually the same and you may differ.]

[0018]

[Formula 4]



[0019] R shows the alkyl group or phenyl group of carbon numbers 1-5 among [type, and n is an integer which shows polymerization degree. However, as long as it is in a structurally stable condition until it starts hydrolysis and condensation polymerization, some OR radicals may be permuted by the OH radical.]

Specifically as the above-mentioned 2 organic-functions alkoxysilane, dimethyl dimethoxysilane, dimethyl diethoxysilane, diphenyl diethoxysilane, diphenyl dimethoxysilane, methylphenyl diethoxysilane, methylphenyl dimethoxysilane, diethyl diethoxysilane, diethyl dimethoxysilane, etc. are mentioned.

[0020] Specifically as the above-mentioned 3 organic-functions alkoxysilane, methyl trimethoxysilane, methyl triethoxysilane, ethyl trimethoxysilane, ethyltriethoxysilane, phenyl trimethoxysilane, phenyl triethoxysilane, etc. are mentioned.

[0021] Specifically as the above-mentioned 4 organic-functions alkoxysilane, a tetramethoxy silane, a tetraethoxy silane, etc. are mentioned.

[0022] transparent and colorless, although it is desirable that polymerization degree is below ten (polymerization degree describes the thing of n as n **** below.) as for the oligomer of the alkoxysilane expressed above (** 4) -- if liquefied, it will not be limited to this. The oligomer of the above-mentioned alkoxysilane does not need to be a compound with this uniform polymerization degree, distribution of polymerization degree may exist or the molecular structure may be intermingled by the shape of a chain, the letter of branching, and annular. If the stability as matter and the reaction time for producing a gel compound are taken into consideration, the thing of 2 - a hexamer is the most desirable. R in the oligomer of the above-mentioned alkoxysilane expresses an alkyl group and a phenyl group, and a methyl group (CH₃) and its ethyl group (C two H₅) are the most desirable especially. In the case of the oligomer of a methoxy silane, when average molecular weight is OGORIMA of 250-700, and an ethoxy silane, specifically, the oligomer of 300-900 has the most desirable average molecular weight.

[0023] In order to hydrolyze the above-mentioned alkoxysilane efficiently and to perform condensation polymerization, it is desirable to add a catalyst beforehand to the system of reaction containing alkoxysilane. As the above-mentioned catalyst, basic catalysts, such as an acid catalyst of a hydrochloric acid, a citric acid, a nitric acid, a sulfuric acid, ammonium fluoride, etc. and ammonia, and a piperidine, are mentioned, for example.

[0024] Furthermore, in order to obtain a uniform gel compound, it is desirable to use mixed liquor with the solvent which has compatibility in water and water and dissolves the above-mentioned alkoxysilane in the above-mentioned system of reaction. As the above-mentioned solvent, alcohol and acetones, such as a methanol, ethanol, isopropanol, and a butanol, an acetonitrile, etc. are mentioned, for example. Alcohol is the most desirable when alcohol's generating at hydrolysis of the generation process of a gel compound and a polymerization reaction and the supercritical drying mentioned later are taken into consideration.

[0025] The fiber object 1 which constitutes the aerogel panel of this invention has adiathermic, and organic high molecular compounds, such as glass fiber, ceramic fiber, polyester, and a polyamide, are mentioned as the quality of the material. These fiber object 1 is used after fiber has only distributed, the multilayer object of a nonwoven fabric, the aggregate of massive fiber, and. By using the fiber object 1 for this core material, the reinforcement of aerogel 2 is reinforced, and aerogel 2 breaks in the case of handling, or it prevents being destroyed. Furthermore, adiathermic [of aerogel] is maintainable. The class of fiber object 1 used as the above-mentioned core material, a configuration, a consistency, thickness, etc. are suitably chosen from the physical properties which the aerogel panel obtained needs.

[0026] Next, the manufacture approach for obtaining the above-mentioned aerogel panel is explained. In order to obtain the above-mentioned aerogel panel, after making the sol-like reaction mixture 3 containing alkoxysilane, the above-mentioned solvent, etc. adhere to the above-mentioned fiber object 1 and gelling it,

supercritical drying is carried out and it produces.

[0027] An example which obtains the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) is shown. The nonwoven fabric 11 which has translucency as a fiber object 1 is used. For thickness, 0.1-0.5mm and a consistency are [the above-mentioned nonwoven fabric 11] 0.1 - 2.0 g/cm³. It is desirable. The translucency of the above-mentioned nonwoven fabric 11 is suitably chosen by the application of an aerogel panel. As shown in drawing 2 , after covering the bottom of a container 4 with one nonwoven fabric 11a and slushing the above-mentioned sol-like reaction mixture 3a on this nonwoven fabric 11a Before this slushed sol-like reaction mixture 3 gels, nonwoven fabric 11b is floated on the top face of this sol-like reaction mixture 3, and after the above-mentioned sol-like reaction mixture 3a which carried out the style gels, sol-like reaction mixture 3b is slushed on nonwoven fabric 11b of the 2nd sheet from a bottom. By repeating until the thickness of a request of this is obtained, the layered product which sol-like reaction mixture 3 gelled between the layers of a nonwoven fabric 11 is obtained. If supercritical drying of this layered product is carried out, the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) will be obtained.

[0028] Moreover, as shown in drawing 3 , the layered product which sol-like reaction mixture 3 gelled between the layers of a nonwoven fabric 11 is obtained by opening two or more sheets and spacing 5 in the bottom of a container 4, installing a nonwoven fabric 11 in it in the cross direction, and making this spacing 5 slush and gel the above-mentioned sol-like reaction mixture 3. If supercritical drying of this layered product is carried out, the aerogel panel which used as the fiber object 1 the multilayer nonwoven fabric 11 shown in drawing 1 (a) will be obtained. Since the obtained aerogel panel uses the nonwoven fabric 11 which has translucency, its translucency is good, while its reinforcement improves and it maintains adiathermic.

[0029] An example which obtains the aerogel panel which used as the fiber object 1 the massive fiber aggregate 12 shown in drawing 1 (b) is shown. As a fiber object 1, the massive fiber aggregate 12 which has openings, such as glass wool, is used. For the above-mentioned massive fiber aggregate 12, a consistency is 0.005 - 0.5 g/cm³. The range is desirable. As shown in drawing 4 , the massive fiber aggregate 12 of desired magnitude is put into the container 4, the above-mentioned sol-like reaction mixture 3 is slushed in a container 4, and the opening of the above-mentioned massive fiber aggregate 12 is filled up with sol-like reaction mixture 3. After the above-mentioned sol-like reaction mixture 3 gels, if supercritical drying is performed, the aerogel panel which used as the fiber object 1 the massive fiber aggregate 12 shown in drawing 1 (b) will be obtained.

[0030] An example which obtains the aerogel panel used as the fiber object 1 which consists of dispersed fiber 13 shown in drawing 1 (c) is shown. Fiber 13 with a diameter [of 1-100 micrometers] and a die length of about 1-5cm is used. It slushes into a container 4, after carrying out mixed stirring of the above-mentioned sol-like reaction mixture 3 and the above-mentioned fiber 13 beforehand, as shown in drawing 5 . The loadings of the above-mentioned fiber 13 have 5 - 30 desirable % of the weight to the aerogel panel obtained. After the above-mentioned sol-like reaction mixture 3 gels, if supercritical drying is performed, the aerogel panel used as the fiber object 1 which consists of dispersed fiber 13 shown in drawing 1 (c) will be obtained.

[0031] When hydrophobing processing is performed in the case of supercritical drying before the above-mentioned supercritical drying, degradation of the dimensional change by moisture absorption of an aerogel panel etc. can be prevented, and it is desirable. The above-mentioned hydrophobing processing permutes the hydrophilic group on the front face of a silica by the hydrophobic group, and uses the hydrophobing processing agent which has the functional group which reacts to a silanol group, and a hydrophobic group. Organic silane compounds, such as hexamethyldisilazane, hexa methyl disiloxane, trimethylchlorosilane, a trimethyl methoxy silane, a trimethyl ethoxy silane, a triethyl ethoxy silane, a triethyl methoxy silane, dimethyldichlorosilane, dimethyl diethoxysilane, methyltrichlorosilane, and ethyl trichlorosilane, are mentioned, and, specifically, as for the above-mentioned hydrophobing processing agent, organic compounds, such as carboxylic acids, such as an acetic acid, formic acid, and a succinic acid, and alkyl halides, such as methyl chloride, are mentioned besides this. A hydrophobing processing agent may use only one sort and may use two or more sorts.

[0032] As a solvent used in case supercritical drying of the fiber object 1 to which the above-mentioned gel compound adhered is carried out, independent or two or more sorts of mixed stock, such as ethanol, a methanol, a dichlorodifluoromethane, a carbon dioxide, and water, are mentioned, for example.

[0033] When using the above-mentioned solvent independently, generally, the gel compound which performed the solvent permutation is put into the above-mentioned solvent and the same solvent together into proof-pressure containers, such as an autoclave, the inside of a container is raised to the temperature more than the

critical point of this solvent, and a pressure, the solvent contained in the gel compound is removed gradually, and, finally desiccation is ended by [of the condition of ordinary temperature ordinary pressure] returning. When using the above-mentioned solvent by two or more sorts of mixing, the solvent contained in a gel compound dissolves in the solvent mixed within the proof-pressure container. The above-mentioned gel compound is made to coexist in the same solvent as the approach of raising a pressure and temperature so that it may be in a supercritical condition in the state of 1 phase, and the solvent contained in a gel compound within a proof-pressure container. Since the solvent in a gel compound is permuted by another soluble high solvent in the state of high pressure and the permutation of a solvent is completed mostly, it changes into the supercritical condition of the above-mentioned solvent, and there is a method of removing the solvent contained in the gel compound etc.

[Translation done.]

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OPERATION

[Function] The fiber object 1 reinforces the reinforcement of aerogel 2 as a core material, aerogel 2 breaks in the case of handling, or it prevents being destroyed while it maintains adiathermic, since the aerogel panel concerning claim 1 of this invention consists of aerogel 2 which has the silica frame to which the fiber object 1 which has adiathermic as a core material, and this fiber object 1 adhered.

[Translation done.]

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EXAMPLE

[Example]

The oligomer (COL coat incorporated company make: methyl silicate 51, average molecular weight 470) of a tetramethoxy silane was used as example 1 alkoxysilane, and 0.01 mols [/l.] aqueous ammonia was used as a solvent as ethanol (reagent chemicals by Nakarai Tesuku, Inc.), water, and a catalyst. The sol-like reaction mixture which blended 120 mols and water by 20 mols, and blended [the oligomer of the above-mentioned tetramethoxy silane] aqueous ammonia for one mol and ethanol by the ratio of 2.16 mols was obtained.

[0036] As a fiber object, the nonwoven fabric (the Kureha tech incorporated company make: die nak S-020) of 0.15mm in thickness and specific gravity 1.08 was used. The bottom of a container was covered with one sheet of this nonwoven fabric, and the above-mentioned sol-like reaction mixture was slushed. After it floated the nonwoven fabric of the 2nd sheet on the sol-like reaction mixture slushed after [of abbreviation] 2 minutes and previous sol-like reaction mixture gelled, sol-like reaction mixture was slushed on the nonwoven fabric of the 2nd sheet. Floated the nonwoven fabric of the 3rd sheet on the sol-like reaction mixture slushed after [of abbreviation] 2 minutes, it was made to put and gel at a room temperature, and the layered product of a gel compound and a nonwoven fabric was obtained. The thickness of this layered product was 8mm.

[0037] Next, a proof-pressure container is used and they are 18 degrees C and the pressure of 55kg/cm² about this layered product. It put in into the liquefaction carbon dioxide and actuation of permuting the ethanol in a gel compound by the carbon dioxide was performed for 3 hours. The temperature of 80 degrees C and the pressure of 160kg/cm² which are the supercritical conditions of a carbon dioxide about the inside of a proof-pressure container after that It carried out and solvent removal was performed for 48 hours. Add hexamethyldisilazane in the ambient atmosphere of this supercritical condition at a rate of 0.3 mols/l. as a hydrophobing processing agent, and it was made to diffuse a hydrophobing processing agent in supercritical fluid over 2 hours, into this supercritical fluid, the gel compound was left and hydrophobing was given. Then, it decompressed, after circulating the carbon dioxide of a supercritical condition, and the ethanol and the hydrophobing processing agent which are contained in a gel compound were removed. The time amount from a hydrophobing processing agent injection to reduced pressure required 15 hours. Then, it took out from the proof-pressure container and the aerogel panel was obtained. this aerogel panel -- thickness -- 8mm and bulk density -- 0.086 g/cm³ it was .

[0038] The aerogel panel was obtained like the example 1 except having obtained the layered product with a thickness of 8.0mm which made three layers the gel compound of example 2 example 1, and made the nonwoven fabric four layers. this aerogel panel -- thickness -- 8mm and bulk density -- 0.115 g/cm³ it was .

[0039] As an example 3 fiber object, they are 0.8mm in thickness, and consistency 0.01 g/cm³. Glass wool (NIPPON MUKI [CO., LTD.] make: super fine SPF-210) was used. After putting this glass wool into a container and slushing the same sol-like reaction mixture as an example 1, it was made to put and gel at a room temperature. Supercritical drying was performed on the same conditions as an example 1 after that, and the aerogel panel was obtained. this aerogel panel -- thickness -- 8mm and bulk density -- 0.055 g/cm³ it was .

[0040] In example 4 example 3, the aerogel panel was obtained like the example 3 except having not performed hydrophobing processing on the occasion of supercritical drying. this aerogel panel -- thickness -- 8mm and bulk density -- 0.051 g/cm³ it was .

[0041] The oligomer (COL coat incorporated company make: methyl silicate 51, average molecular weight 470) of a tetramethoxy silane was used as example 5 alkoxysilane, and 0.01 mols [/l.] aqueous ammonia was used as a solvent as ethanol (reagent by Nakarai Tesuku, Inc.), water, and a catalyst. The sol-like reaction mixture which blended 50 mols and water by 20 mols, and blended [the oligomer of the above-mentioned

tetramethoxy silane] aqueous ammonia for one mol and ethanol by the ratio of 0.22 mols was obtained. The aerogel panel was obtained like the example 3 except having used the above-mentioned sol-like reaction mixture. this aerogel panel -- thickness -- 8mm and bulk density -- 0.121 g/cm³ it was .

[0042] The same sol-like reaction mixture as example 6 example 1 was used. Fiber with a die length of 20mm was mixed and stirred at a rate of the 0.1 weight section and the above-mentioned sol-like reaction mixture 14 weight section, and it slushed into the container. Supercritical drying was carried out on the same conditions as an example 1 after gelation, and the aerogel panel was obtained. The rate of the above-mentioned fiber became 12 % of the weight to the obtained aerogel panel. moreover, this aerogel panel -- thickness -- 8mm and bulk density -- 0.051 g/cm³ it was .

[0043] After obtaining a gel compound using the same sol-like reaction mixture as example of comparison 1 example 1, supercritical drying was carried out on the same conditions as an example 1, and aerogel was obtained. this aerogel -- thickness -- 8mm and bulk density -- 0.045 g/cm³ it was .

After obtaining a gel compound using the same sol-like reaction mixture as example of comparison 2 example 5, supercritical drying was carried out on the same conditions as an example 5, and aerogel was obtained. this aerogel -- thickness -- 8mm and bulk density -- 0.085 g/cm³ it was .

[0044] It evaluated the aerogel panel of the acquired examples 1-6, the reinforcement of the aerogel of the examples 1-2 of a comparison, and adiathermic. Flexural strength was measured as reinforcement. Thermal conductivity was measured as adiathermic. The above-mentioned thermal conductivity measured the thermal conductivity of 20 degrees C - 40 degrees C based on ASTM-C518 using the Hidehiro energy machine incorporated company make and thermal conductivity measuring apparatus.

[0045] As for the example, adiathermic was maintained and it was checked that reinforcement is improving as the result was shown in Table 1.

[0046]

[Table 1]

	曲げ強度 (kg/ca ²)	熱伝導率 (Kcal/mh°C)
実施例 1	2.40	0.014
実施例 2	3.18	0.015
実施例 3	1.86	0.016
実施例 4	1.77	0.016
実施例 5	3.56	0.014
実施例 6	0.32	0.013
比較例 1	0.05	0.013
比較例 2	0.09	0.010

[0047] The light transmittance of the aerogel panel of the acquired examples 1, 2, and 6 and the aerogel of the examples 1-2 of a comparison was measured. The above-mentioned light transmittance measured the spectral distribution of a light region, and asked for light permeability based on JIS-R3106. It was checked by the result as Table 2 that the aerogel panel of examples 1, 2, and 6 has translucency.

[0048]

[Table 2]

	光透過率 (%)
実施例 1	7 0
実施例 2	5 0
実施例 6	8 1
比較例 1	9 2
比較例 2	9 3

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] (a) is the perspective view of the aerogel panel concerning one example of this invention, (b) is the perspective view of the aerogel panel concerning other examples of this invention, and (c) is the perspective view of the aerogel panel concerning other examples of this invention.

[Drawing 2] It is the schematic diagram having shown an example of a process which obtains the aerogel panel of this invention.

[Drawing 3] It is the schematic diagram having shown an example of a process which obtains the aerogel panel of this invention.

[Drawing 4] It is the schematic diagram having shown an example of a process which obtains the aerogel panel of this invention.

[Drawing 5] It is the schematic diagram having shown an example of a process which obtains the aerogel panel of this invention.

[Description of Notations]

- 1 Fiber Object
- 2 Aerogel
- 3 Sol-like Reaction Mixture

[Translation done.]

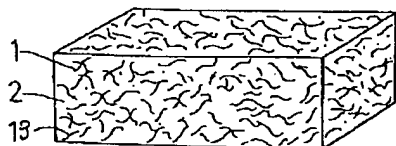
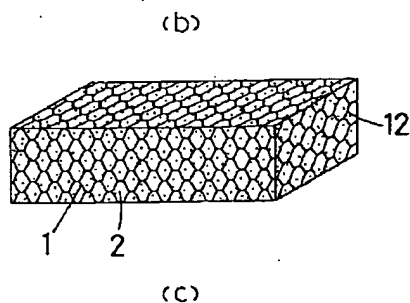
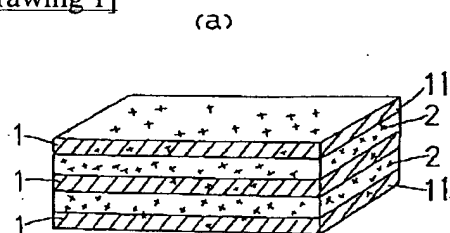
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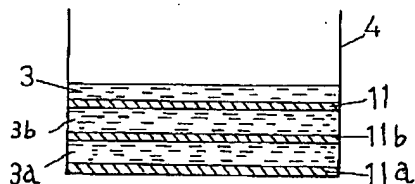
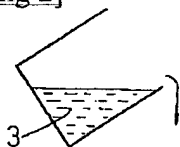
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DRAWINGS

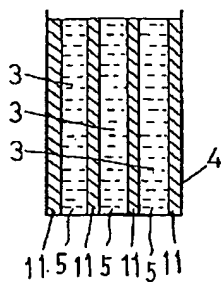
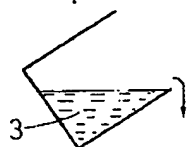
[Drawing 1]



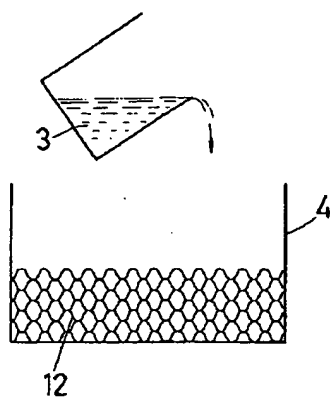
[Drawing 2]



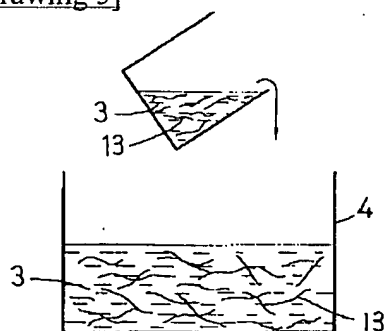
[Drawing 3]



[Drawing 4]



[Drawing 5]



[Translation done.]

PAT-NO: JP408034678A
DOCUMENT-IDENTIFIER: JP 08034678 A
TITLE: AEROGEL PANEL
PUBN-DATE: February 6, 1996

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COUNTRY
N/A

APPL-NO: JP06175839
APPL-DATE: July 27, 1994

INT-CL (IPC): C04B038/00, C08J005/04

ABSTRACT:

PURPOSE: To obtain an aerogel panel with improved strength having translucency while maintaining heat insulating property by using a fiber body having heat insulating property as the core material and depositing an aerogel having a silica skeleton on the core material.

CONSTITUTION: As for the fiber material 1 having heat insulating property as the core material, a multilayered nonwoven fabric (a) having translucency, an aggregated fiber block (b) having voids or dispersed fibers (c) is used. The core is impregnated with an alkoxysilane sol comprising alkoxysilane, water and alcohol. After the sol is changed into a gel, the material is dried under supercritical conditions higher than the critical point of the solvent to produce an aerogel. Thus, the aerogel 2 having a silica skeleton deposits on the fiber 1, and thereby, an aerogel panel in such a state that the aerogel 2 is reinforced with the fiber material 1 is obtd.

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DERWENT-ACC-NO: 1996-145349

DERWENT-WEEK: 199615

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TITLE: Heat insulating silica aero-gel panels, for acoustic material, etc. - comprises heat insulating core of fibre and silica aero gel deposited on the fibres

PATENT-ASSIGNEE: MATSUSHITA ELECTRIC WORKS LTD(MATW)

PRIORITY-DATA: 1994JP-0175839 (July 27, 1994)

PATENT-FAMILY:

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C04B 038/00			

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
JP 08034678A	N/A	1994JP-0175839	July 27,
1994			

INT-CL (IPC): C04B038/00, C08J005/04 , C08L083:00

ABSTRACTED-PUB-NO: JP 08034678A

BASIC-ABSTRACT:

A aero-gel panel comprises heat-insulating core material (1) made of fibres and silica aero-gel (2) which is deposited on the fibres.

USE - Used as heat-insulating material acoustic material, or catalyst supports.

ADVANTAGE - The panels are heat-insulating, having good mechanical strength.

CHOSEN-DRAWING: Dwg.1/5

TITLE-TERMS: HEAT INSULATE SILICA AERO GEL PANEL ACOUSTIC MATERIAL COMPRISE
HEAT INSULATE CORE FIBRE SILICA AERO GEL DEPOSIT FIBRE

DERWENT-CLASS: J04 L02

CPI-CODES: J04-E03; L02-D15;

UNLINKED-DERWENT-REGISTRY-NUMBERS: 0245S; 1694P

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1996-045940

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特開平8-34678

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C 0 8 J 5/04	C F H			
// C 0 8 L 83:00				

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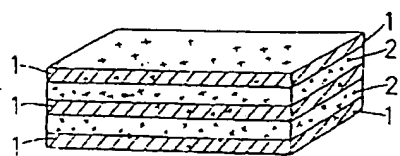
(54) 【発明の名称】 エアロゲルパネル

(57) 【要約】

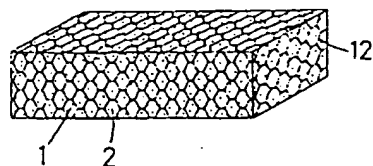
【目的】 断熱性を維持し、強度の向上したエアロゲルパネルを提供する。

【構成】 芯材として断熱性を有する繊維体1と、この繊維体1に付着されたシリカ骨格を有するエアロゲル2とからなる。

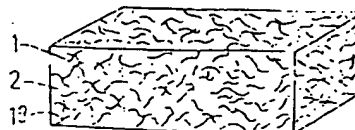
(a)



(b)



(c)



1

【特許請求の範囲】

【請求項1】 芯材として断熱性を有する繊維体(1)と、この繊維体(1)に付着されたシリカ骨格を有するエアロゲル(2)からなることを特徴とするエアロゲルパネル、

【請求項2】 上記繊維体(1)が透光性を有する多層の不織布からなることを特徴とする請求項1記載のエアロゲルパネル、

【請求項3】 上記繊維体(1)が空隙を有する塊状繊維集合体からなることを特徴とする請求項1記載のエアロゲルパネル、

【請求項4】 上記繊維体(1)が分散した繊維からなることを特徴とする請求項1記載のエアロゲルパネル、

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は断熱性を有するエアロゲルパネルに関するものである。

【0002】

【従来の技術】断熱材料としてシリカ骨格を有するエアロゲルが知られている。このエアロゲルは、米国特許第4402927号、米国特許第4432956号、米国特許第4610863号に開示されている如く、アルコキシシラン(別にシリコンアルコキシド、アルキルシリケートとも称する)を加水分解し、縮重合して得られる、シリカ骨格からなる湿潤状態のゲル状化合物をアルコール、または液化二酸化炭素等の溶媒の存在下で、この溶媒の臨界点以上の超臨界条件で乾燥することによって得られる。このエアロゲルは透光性を有する断熱材として有用である。ところが、これらの方法で得られたエアロゲルは、非常に軽量であると共に、強度が小さく脆いため、割れたり壊れ易く、取扱いが難しい欠点がある。

【0003】

【発明が解決しようとする課題】本発明は上述の事実を鑑みてなされたもので、その目的とするところは、断熱性を維持し、強度の向上したエアロゲルパネルを提供することにある。

【0004】さらに、他の目的とするところは、透光性を有するエアロゲルパネルを提供することにある。

【0005】

【課題を解決するための手段】本発明の請求項1に係るエアロゲルパネルは、芯材として断熱性を有する繊維体1と、この繊維体1に付着されたシリカ骨格を有するエアロゲル2からなることを特徴とする。

【0006】本発明の請求項2に係るエアロゲルパネルは、請求項1記載のエアロゲルパネルにおいて、上記繊維体1が透光性を有する多層の不織布からなることを特徴とする。

【0007】本発明の請求項3に係るエアロゲルパネルは、請求項1記載のエアロゲルパネルにおいて、上記繊維

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繊維1が空隙を有する塊状繊維集合体からなることを特徴とする。

【0008】本発明の請求項4に係るエアロゲルパネルは、請求項1記載のエアロゲルパネルにおいて、上記繊維体1が分散した繊維からなることを特徴とする。

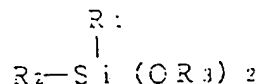
【0009】以下本発明を図面に基づいて詳細に説明する。図1(a)(b)(c)は本発明の一実施例に係るエアロゲルパネルの斜視図であり、図2、図3、図4、及び図5はエアロゲルパネルを得る製法の一例を示した概略図である。

【0010】本発明のエアロゲルパネルは、芯材として繊維体1と、この繊維体1に付着されたシリカ骨格を有するエアロゲル2とからなる。上記エアロゲル2は、アルコキシシラン化合物を加水分解し、縮重合して得られたシリカ骨格からなる湿潤状態のゲル状化合物をアルコール、または液化二酸化炭素等の溶媒の存在下で、この溶媒の臨界点以上の超臨界条件で乾燥することによって得られる多孔質材料である。

【0011】本発明に用いられる上記アルコキシシラン化合物としては、下式(化1)で表される2官能アルコキシシラン、下式(化2)で表される3官能アルコキシシラン、下式(化3)で表される4官能アルコキシシラン、及び、下式(化4)で表されるアルコキシシランのオリゴマーが挙げられる。

【0012】

【化1】



【0013】〔式中、 R_1 、 R_2 、 R_3 は、互いに独立に、炭素数1～5のアルキル基又はフェニル基を示す、2個の R_3 は互いに同じであってよく、異なってもよい。〕

【0014】

【化2】



【0015】〔式中、 R_4 、 R_5 は、互いに独立に、炭素数1～5のアルキル基又はフェニル基を示す、3個の R_5 は互いに同じであってよく、異なってもよい。〕

【0016】

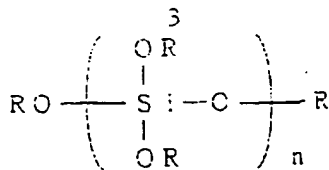
【化3】



【0017】〔式中、 R_6 は炭素数1～5のアルキル基又はフェニル基を示す、4個の R_6 は互いに同じであってよく、異なってもよい。〕

【0018】

【化4】



【0019】〔式中、Rは炭素数1～5のアルキル基又はフェニル基を示し、nは重合度を示す整数である。但し、加水分解、縮重合を開始するまで構造的に安定な状態であれば、一部のOR基がOH基に置換されていても構わない。〕

上記2官能アルコキシシランとしては、具体的には、ジメチルジメトキシシラン、ジメチルジエトキシシラン、ジフェニルジエトキシシラン、ジフェニルジメトキシシラン、メチルフェニルジエトキシシラン、メチルフェニルジメトキシシラン、ジエチルジエトキシシラン、ジエチルジメトキシシラン等が挙げられる。

【0020】上記3官能アルコキシシランとしては、具体的には、メチルトリメトキシシラン、メチルトリエトキシシラン、エチルトリメトキシシラン、エチルトリエトキシシラン、フェニルトリメトキシシラン、フェニルトリエトキシシラン等が挙げられる。

【0021】上記4官能アルコキシシランとしては、具体的には、テトラメトキシシラン、テトラエトキシシラン等が挙げられる。

【0022】前記(化4)で表されるアルコキシシランのオリゴマーは、重合度が10(以下重合度がnのものはn量体と記す。)以下であることが好ましいが、無色透明な液状であれば、これに限定されない。上記アルコキシシランのオリゴマーは、この重合度が均一な化合物である必要はなく、重合度の分布が存在したり、分子構造が鎖状、分岐状、及び環状で混在していても構わない。物質としての安定性や、ゲル状化合物を作製するための反応時間を考慮すれば、2～6量体のものが最も好ましい。上記アルコキシシランのオリゴマー内のRはアルキル基、フェニル基を表し、中でも、メチル基(CH₃)、エチル基(C₂H₅)が最も好ましい。具体的には、メトキシシランのオリゴマーの場合には平均分子量が250～700、エトキシシランのオリゴマーの場合には平均分子量が300～900のオリゴマーが最も好ましい。

【0023】上記アルコキシシランを効率良く加水分解し、縮重合を行うためには、アルコキシシランを含む反応系に予め触媒を添加しておくことが好ましい。上記触媒としては、例えば、塩酸、クエン酸、硝酸、硫酸、フッ化アンモニウム等の酸性触媒、及び、アンモニア、ヒペリジン等の塩基性触媒が挙げられる。

【0024】さらに、均一なゲル状化合物を得るために、上記反応系には、水と、水に相溶性を有し、且つ上記アルコキシシランを溶解する溶媒との混合液を用いることが好ましい。上記溶媒としては、例えばメタノー

ル、エタノール、イソプロパノール、ブタノール等のアルコールやアセトン、アセトニトリル等が挙げられる。ゲル状化合物の生成過程の加水分解、重合反応でアルコールが生成すること、及び後述する超臨界乾燥を考慮すると、アルコールが最も好ましい。

【0025】本発明のエアロゲルパネルを構成する繊維体1は断熱性を有し、材質としては、ガラスファイバー、セラミックファイバー、ポリエステル、ポリアミド等の有機高分子化合物が挙げられる。これら繊維体1は不織布の多層体、塊状繊維の集合体、単に繊維が分散した状態で用いられる。この芯材に繊維体1を用いることにより、エアロゲル2の強度を補強し、取扱いの際にエアロゲル2が割れたり、破壊されることを防止する。さらに、エアロゲルの断熱性を維持することができる。上記芯材となる繊維体1の種類、形状、密度、厚み等は得られるエアロゲルパネルの必要とする物性から適宜選択される。

【0026】次に、上記エアロゲルパネルを得るための製造方法について説明する。上記エアロゲルパネルを得るには、アルコキシシラン、上記溶媒等を含むゾル状反応液3を上記繊維体1に付着させ、ゲル化した後に超臨界乾燥して作製する。

【0027】図1(a)に示す多層の不織布11を繊維体1としたエアロゲルパネルを得る一例を示す。繊維体1として透光性を有する不織布11を用いる。上記不織布11は厚さが0.1～0.5mm、密度が0.1～2.0g/cm³が好ましい。上記不織布11の透光性はエアロゲルパネルの用途により適宜選択される。図2に示す如く、容器4の底に不織布11aを1枚敷き、この不織布11aの上に上記ゾル状反応液3aを流し込んだ後に、この流し込んだゾル状反応液3aがゲル化する前にこのゾル状反応液3の上面に不織布11bを浮かべ、上記流し込んだゾル状反応液3aがゲル化した後に、底から2枚目の不織布11bの上にゾル状反応液3bを流し込む、これを所望の厚みが得られるまで繰り返すことにより、不織布11の層間にゾル状反応液3がゲル化した積層体を得られる。この積層体を超臨界乾燥すると、図1(a)に示す多層の不織布11を繊維体1としたエアロゲルパネルが得られる。

【0028】また、図3に示す如く、容器4の底にクロス方向に不織布11を複数枚、間隔を開けて設置し、この間隔に上記ゾル状反応液3を流し込みゲル化させることにより、不織布11の層間にゾル状反応液3がゲル化した積層体を得られる。この積層体を超臨界乾燥すると、図1(a)に示す多層の不織布11を繊維体1としたエアロゲルパネルが得られる。得られたエアロゲルパネルは強度が向上し、断熱性を維持すると共に、透光性を有する不織布11を用いるので透光性が良好である。

【0029】図1(b)に示す塊状繊維集合体12を繊

繊維1としたエアロゲルパネルを得る一例を示す。繊維体1として、グラスウール等の空隙を有する塊状繊維集合体12を用いる。上記塊状繊維集合体12は、密度が $0.005 \sim 0.5 \text{ g/cm}^3$ の範囲が好ましい、図4に示す如く、容器4に所望の大きさの塊状繊維集合体12を入れておき、容器4内に上記ゾル状反応液3を流し込み、上記塊状繊維集合体12の空隙にゾル状反応液3を充填する。上記ゾル状反応液3がゲル化した後に、超臨界乾燥を行うと図1(b)に示す塊状繊維集合体12を繊維体1としたエアロゲルパネルが得られる。

【0030】図1(c)に示す分散した繊維13からなる繊維体1としたエアロゲルパネルを得る一例を示す。直径 $1 \sim 100 \mu\text{m}$ 、長さ $1 \sim 5 \text{ cm}$ 程度の繊維13が用いられる。図5に示す如く、上記ゾル状反応液3と上記繊維13を予め混合攪拌した後に、容器4に流し込む。上記繊維13の配合量は得られるエアロゲルパネルに対し、 $5 \sim 30$ 重量%が好ましい、上記ゾル状反応液3がゲル化した後に、超臨界乾燥を行うと図1(c)に示す分散した繊維13からなる繊維体1としたエアロゲルパネルが得られる。

【0031】上記超臨界乾燥の前、又は超臨界乾燥の際に、疎水化処理を行うと、エアロゲルパネルの吸湿による寸法変化等の劣化を防止でき、好ましい。上記疎水化処理は、シリカ表面の親水基を疎水基で置換するもので、シラノール基に対して反応する官能基と疎水基を有する疎水化処理剤を用いる。上記疎水化処理剤は、具体的には、ヘキサメチルジシラザン、ヘキサメチルジシロキサン、トリメチルクロロシラン、トリメチルメトキシシラン、トリメチルエトキシシラン、トリエチルメトキシシラン、ジメチルジクロロシラン、ジメチルジエトキシシラン、メチルトリクロロシラン、エチルトリクロロシラン等の有機シラン化合物が挙げられ、これ以外にも、酢酸、硝酸、コハク酸等のカルボン酸や、メチルクロリド等のハロゲン化アルキル等の有機化合物が挙げられる。疎水化処理剤は1種のみを用いてもよいし、2種以上を用いてもよい。

【0032】上記ゲル状化合物が付着した繊維体1を超臨界乾燥させる際に用いる溶媒としては、例えば、エタノール、メタノール、ジクロロジフルオロメタン、二酸化炭素、水等の単独又は2種以上の混合系が挙げられる。

【0033】上記溶媒を単独で用いる場合は、一般には、上記溶媒と、同一の溶媒に溶媒置換を行ったゲル状化合物をオートクレーブ等の耐圧容器の中に一緒に入れ、容器内をこの溶媒の臨界点以上の温度、圧力まで上昇させ、ゲル状化合物に含まれている溶媒を徐々に除去し、最終的に常温常圧の状態に戻すことにより乾燥を終了する。上記溶媒を2種以上の混合で用いる場合は、例えば、耐圧容器内で混合した溶媒にゲル状化合物に含まれる溶媒が溶解し、一相状態で超臨界状態になるように

圧力、温度を上昇させる方法、耐圧容器内でゲル状化合物に含まれる溶媒と同一の溶媒内に上記ゲル状化合物を併存させ、高压状態でゲル状化合物中の溶媒を溶解性の高い別の溶媒に置換し、ほぼ溶媒の置換を完結させてから、上記溶媒の超臨界状態にして、ゲル状化合物に含まれている溶媒を除去する方法等がある。

【0034】

【作用】本発明の請求項1に係るエアロゲルパネルは、芯材として断熱性を有する繊維体1と、この繊維体1に付着されたシリカ骨格を有するエアロゲル2からなるので、断熱性を維持すると共に、繊維体1が芯材としてエアロゲル2の強度を補強し、取扱いの際にエアロゲル2が割れたり、破壊されることを防止する。

【0035】

【実施例】

実施例1

アルコキシシランとしてテトラメトキシシランのオリゴマー（コルコード株式会社製：メチルシリケート51、平均分子量470）、溶媒としてエタノール（ナカライテスク株式会社製特級試薬）、水、及び触媒として0.01モル/リットルのアンモニア水を用いた、上記テトラメトキシシランのオリゴマーを1モル、エタノールを120モル、水を20モル、アンモニア水を2.16モルの比率で配合したゾル状反応液を得た。

【0036】繊維体として、厚さ0.15mm、比重1.08の不織布（呉羽テック株式会社製：ダイナックS-020）を用いた。容器の底にこの不織布を1枚敷き、上記ゾル状反応液を流し込んだ。略2分後に流し込んだゾル状反応液の上に2枚目の不織布を浮かべ、先のゾル状反応液がゲル化した後に、2枚目の不織布の上にゾル状反応液を流し込んだ。略2分後に流し込んだゾル状反応液の上に3枚目の不織布を浮かべ、室温で静置し、ゲル化させ、ゲル状化合物と不織布の積層体を得た。この積層体の厚みは8mmであった。

【0037】次に、耐圧容器を用い、この積層体を18℃、圧力 5 kg/cm^2 の液化二酸化炭素中に入れ、ゲル状化合物内のエタノールを二酸化炭素に置換する操作を3時間行った。その後耐圧容器内を二酸化炭素の超臨界条件である温度 80°C 、圧力 160 kg/cm^2 とし、溶媒除去を48時間行った。この超臨界状態の雰囲気には、疎水化処理剤としてヘキサメチルジシラザンを用い、3モル/リットルの割合で添加し、2時間かけて疎水化処理剤を超臨界流体中に拡散させ、この超臨界流体中にゲル状化合物を放置し疎水化を施した。その後、超臨界状態の二酸化炭素を流通した後に減圧し、ゲル状化合物に含まれるエタノールと疎水化処理剤を除去した。疎水化処理剤投入から減圧までの時間は15時間を要した。その後、耐圧容器から取り出しエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が 0.08 g/cm^3 であった。

【0038】実施例2

実施例1のゲル状化合物を3層、不織布を4層とした厚さ8.0mmの積層体を得た以外は実施例1と同様にしてエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が0.115 g/cm³であった。

【0039】実施例3

繊維体として、厚さ0.8mm、密度0.01 g/cm³のグラスウール（日本無機株式会社製：スーパーファインSPF-210）を用いた。容器にこのグラスウールを入れ、実施例1と同様のゾル状反応液を流し込んだ後、室温で静置し、ゲル化させた。その後実施例1と同様の条件で超臨界乾燥を行いエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が0.055 g/cm³であった。

【0040】実施例4

実施例3において、超臨界乾燥の際に疎水化処理を行わなかった以外は実施例3と同様にしてエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が0.051 g/cm³であった。

【0041】実施例5

アルコキシシランとしてテトラメトキシシランのオリゴマー（コルコート株式会社製：メチルシリケート51、平均分子量470）、溶媒としてエタノール（ナカライテスク株式会社製試薬）、水、及び触媒として0.01モル リットルのアンモニア水を用いた。上記テトラメトキシシランのオリゴマーを1モル、エタノールを50モル、水を20モル、アンモニア水を0.22モルの比率で配合したゾル状反応液を得た。上記ゾル状反応液を用いた以外は実施例3と同様にしてエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が0.121 g/cm³であった。

【0042】実施例6

実施例1と同様のゾル状反応液を用いた。長さ20mmの繊維を0.1重量部と上記ゾル状反応液1.4重量部の割合で混合、攪拌し、容器に流し込んだ。ゲル化後、実施例1と同様の条件で超臨界乾燥し、エアロゲルパネルを得た。上記繊維の割合は得られたエアロゲルパネルに対し1.2重量%となった。また、このエアロゲルパネルは厚さが8mm、かさ密度が0.051 g/cm³であった。

【0043】比較例1

実施例1と同様のゾル状反応液を用い、ゲル状化合物を得た後に、実施例1と同様の条件で超臨界乾燥しエアロゲルを得た。このエアロゲルは厚さが8mm、かさ密度が0.045 g/cm³であった。

比較例2

実施例5と同様のゾル状反応液を用い、ゲル状化合物を得た後に、実施例5と同様の条件で超臨界乾燥しエアロゲルを得た。このエアロゲルは厚さが8mm、かさ密度

が0.085 g/cm³であった。

【0044】得た実施例1～6のエアロゲルパネル、及び比較例1～2のエアロゲルの強度、及び、断熱性を評価した。強度として曲げ強度を測定した。断熱性として熱伝導率を測定した。上記熱伝導率は、英弘精機株式会社製、熱伝導率測定装置を用い、ASTM-C518に基づいて、20℃～40℃の熱伝導率を測定した。

【0045】結果は表1に示すとおり、実施例は断熱性が維持され、強度が向上していることが確認された。

【0046】

【表1】

	曲げ強度 (kg/cm ²)	熱伝導率 (Kcal/mh℃)
実施例1	2.40	0.014
実施例2	3.13	0.015
実施例3	1.85	0.016
実施例4	1.77	0.016
実施例5	3.56	0.014
実施例6	0.32	0.013
比較例1	0.05	0.013
比較例2	0.99	0.010

【0047】得た実施例1、2、及び6のエアロゲルパネル、及び比較例1～2のエアロゲルの光透過率を測定した。上記光透過率は、可視光域の分光分布を測定し、可視光透過率をJIS-R3106に基づいて求めた。結果は表2のとおり、実施例1、2、及び6のエアロゲルパネルは透光性を有することが確認された。

【0048】

【表2】

	光透過率 (%)
実施例1	70
実施例2	50
実施例6	81
比較例1	32
比較例2	33

【0049】

【発明の効果】本発明の請求項1乃至請求項4に係るエアロゲルパネルは、断熱性を維持し、強度が優れる。

【0050】さらに、本発明の請求項2、又は請求項4に係るエアロゲルパネルは、特に、透光性を有する。

【0051】本発明のエアロゲルパネルは断熱材、音響材料、触媒担持体等の用途に有用である。

【図面の簡単な説明】

【図1】(a)は本発明の一実施例に係るエアロゲルパネルの斜視図であり、(b)は本発明の他の実施例に係るエアロゲルパネルの斜視図であり、(c)は本発明の他の実施例に係るエアロゲルパネルの斜視図である。

【図2】本発明のエアロゲルパネルを得る製法の一例を示した概略図である。

【図3】本発明のエアロゲルパネルを得る製法の一例を示した概略図である。

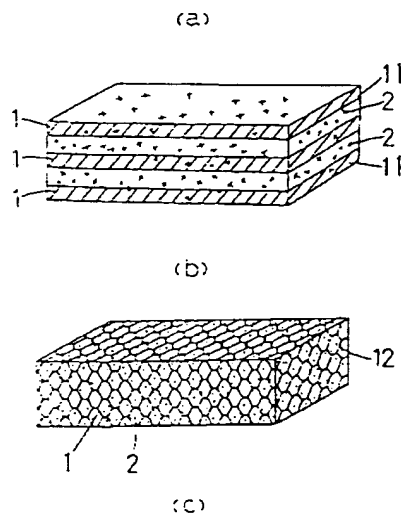
【図4】本発明のエアロゲルパネルを得る製法の一例を示した概略図である。

【図5】本発明のエアロゲルパネルを得る製法の一例を示した概略図である。

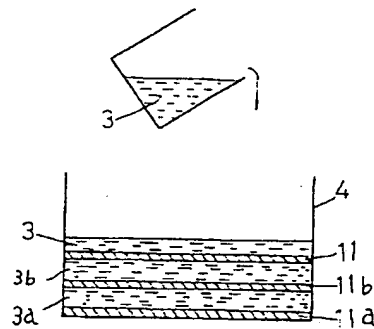
【符号の説明】

- 1 繊維体
- 2 エアロゲル
- 3 ゼル状反応液

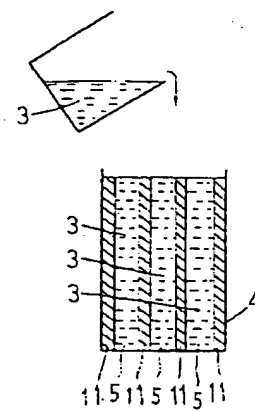
【図1】



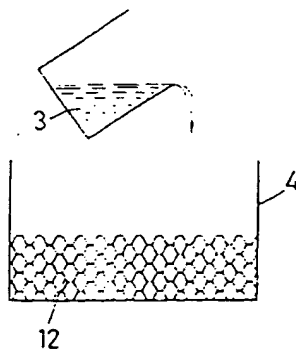
【図2】



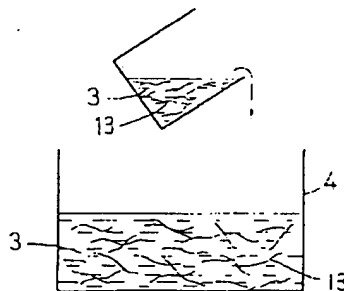
【図3】



【図4】



【図5】



【手続補正書】

【提出日】平成7年2月1日

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】0022

【補正方法】変更

【補正内容】

【0022】前記(化4)で表されるアルコキシシランのオリゴマーは、重合度が10(以下重合度がnのものはn量体と記す。)以下であることが好ましいが、無色透明な液状であれば、これに限定されない、上記アルコキシシランのオリゴマーは、この重合度が均一な化合物である必要はなく、重合度の分布が存在したり、分子構造が鎖状、分岐状、及び環状で混在していても構わない。物質としての安定性や、ゲル状化合物を作製するための反応時間を考慮すれば、2～6量体のものが最も好ましい。上記アルコキシシランのオリゴマー内のRはアルキル基、フェニル基を表し、中でも、メチル基(CH₃)、エチル基(C₂H₅)が最も好ましい。具体的に

は、メトキシシランのオリゴマーの場合には平均分子量が250～700、エトキシシランのオリゴマーの場合には平均分子量が300～900のオリゴマーが最も好ましい。

【手続補正2】

【補正対象書類名】明細書

【補正対象項目名】0039

【補正方法】変更

【補正内容】

【0039】実施例3

繊維体として、密度0.01g/cm³のグラスウール(日本無機株式会社製:スーパーファインSPF-210)を用いた。容器にこのグラスウールを入れ、実施例1と同様のゾル状反応液を流し込んだ後、室温で静置し、ゲル化させた。その後実施例1と同様の条件で超臨界乾燥を行いエアロゲルパネルを得た。このエアロゲルパネルは厚さが8mm、かさ密度が0.055g/cm³であった。

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